

Byron H. Webb

Preparation for Freezing and Freezing of Dairy Products

INTRODUCTION

Dairy products other than ice cream and other frozen desserts require only simple preparation for freezing. Ice cream, ice milk, and sherbets, discussed in Vol. 4, Chap. 21, are complicated mixtures of ingredients and the freezing process plays a very special part in their manufacture. Ice cream is eaten in a frozen state, but other dairy products are frozen only to preserve them for future use. They are thawed before consumption. Freezing of dairy products is a process for maintaining them in a fresh state during necessary periods of storage.

THE FROZEN DAIRY PRODUCTS INDUSTRY

The annual production of milk in the United States is about 120 billion pounds. Somewhat less than half of this is consumed as fluid milk. Part of the remainder is manufactured into products which are not considered perishable but which benefit from cool storage. These include the sterilized and dried milk products. Another portion of the milk is manufactured into cheese and butter which are usually stored at cold temperatures but not necessarily at temperatures at which the water they contain is frozen. The freezing points of some dairy products are given in Table 31, Chap. 10, Vol. 2. The freezing points should not be considered as the temperature

Table 30

Peak Cold Storage Holdings of Certain Dairy Products in U.S. Warehouses 1962-1966¹

Date ¹	Product Held at Indicated Temp. (Million Lb.)			
	Cream ² (0° to -20°F.) (-18° to -29°C.)	Plastic Cream ³ (0° to -20°F.) (-18° to -29°C.)	Butter (0° to -20°F.) (-18° to -29°C.)	Natural Cheese (30° to 33°F.) (-1.1° to 0.6°C.)
1962	13	2	469	527
1963	11	2	401	439
1964	14	2	244	399
1965	15	3	220	415
1966	17	3	92	403
Total U.S. Production 1966			1,119	1,873

Source: U.S. Dept. Agr. Statistical Reporting Service, Annual Statistical Summary.

¹ Peak holdings occur during June to August each year.

² Fat content less than 75%.

³ Fat content greater than 75%.

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that must be reached to preserve the products. In some cases preservation can be satisfactorily achieved by holding at temperatures above those at which ice is formed.

Cream, plastic cream, and butter are listed in the U.S. Dept. of Agr. cold storage reports (Table 30) as commercial products that are held at freezing temperatures. The cream and plastic cream are prepared and frozen from the spring and summer surplus. They are not consumer items but are held for food manufacture during fall and winter when prices of these commodities have advanced. Butter is the common form of milk product used by industry and government to store milkfat. Government purchases of surplus milk products for price support have generally been in the form of butter and nonfat dry milk. Butter held at -10°F. (-23°C.) and nonfat dry milk below 40°F. (4°C.) remain acceptable for 1 to 2 yr.

Fluid milk is sometimes frozen to preserve it for short periods. Its high water content (87%) makes freezing an expensive method of keeping milk. Nevertheless, some milk is frozen in cartons for use by the armed forces, on ships and where supplies of fresh milk are difficult to obtain. The freezing of a 3:1 milk concentrate has been practiced commercially by one company for special market tests. Very recently the freezing of starter cultures has been done on a commercial scale. Freezing is not an entirely satisfactory way to preserve cheese, but cold storage is desirable. Freezing of canned evaporated milk may cause rupturing of the can and subsequent spoilage. Sweetened condensed milk is preserved by sugar, which also lowers its freezing point to about 5°F. (-15°C.). There is no advantage in holding it below this temperature.

Storage Life of Refrigerated Dairy Products

The estimated storage life of dairy products when held under common storage conditions is shown in Table 31 (Foreign Agr. Serv. 1966). Many of the products given in Table 31 are not held in frozen storage, but the life of all of them is prolonged by cold storage. (Table 31 is on p. 320.)

While freezing damages the body of some products, it prolongs the life of others. Static freezing, without agitation, is used for cream, plastic cream, butter, bacteriological cultures, milk, and concentrated milk. Fast freezing in the package sometimes affords greater protection to physical properties but often the differences are not important. Unlike ice cream, these products are not eaten in the frozen state so that large ice crystals and a coarse body may not be objectionable.

Use of Additives

Usually only slight or no modifications are made in the composition or the processing of fluid milk, cream, butter, or starter cultures to prepare

them for freezing. But milk concentrated to the 2:1 or 3:1 level requires special processing if it is to survive frozen storage in acceptable condition.

The industry has tried to avoid the use of additives in the preparation of dairy products for freezing preservation. When additives are used they must conform to state and federal requirements. Products that enter interstate commerce must be labeled in accordance with the Food & Drug Administration's definition and standard of identity. Protection against destructive physical effects of freezing (i.e., gelation in frozen concentrated milk associated with lactose crystallization and improved by lactase additive) is best sought by improvement or modification of processing techniques or by changes in the normal composition of the product rather than through the use of additives.

THE FREEZING OF CREAM

Cream is frozen to preserve it for use in food manufacture, usually in ice cream. Two fat concentrations are common—50% frozen cream and 80% plastic cream. Frozen cream is not prepared in small retail packages but is frozen in bulk for food preparation as a means of taking care of surpluses and shortages. Brown (1963), in a bibliography on frozen cream, has classified the reasons for the freezing of cream into five categories: (1) Cream is frozen in summer to be churned into butter in the winter in Germany and Holland. This is linked with a favorable price structure and it may not be economical elsewhere. Godbersen (1964) points out that cream frozen and held in Germany for 5 to 6 months made better quality butter than was possible with storage of the butter. This would not necessarily be so if the butter were high in quality and the storage was at a constant -20°F . (-29°C .) temperature. (2) In the United States cream is frozen for later use, largely in ice cream manufacture. In this case, the problems of texture and body are not of prime importance because the cream is processed into ice cream mix after thawing. (3) Frozen whipped cream has been considered as a retail product, but it has never attained significant volume. (4) Frozen table cream may be prepared for export or winter use. This is convenient for storing, but it would seem to have little advantage over a high-fat frozen cream. (5) Freezing of cream in any form to penetrate tariff barriers. These may specifically exclude butter and other high-fat dairy products but not cream.

Growth of bacteria in cream is stopped during frozen storage. Counts of most organisms, yeasts, and molds decrease substantially as the period of frozen storage advances.

Frozen Cream

Frozen cream contains 50% fat, in contrast to plastic cream of 80% fat. Pasteurized cream is usually frozen for food manufacture but Aule and Storgårds (1965) describe a method for farm freezing of raw cream that kept well for 7 days at 9° to 0°F. (−13° to −18°C.). It was essential to pasteurize the cream immediately on defrosting.

Flavor Changes in Frozen Cream.—Oxidation of the fat during frozen storage is the principal flavor defect of frozen cream. This must be prevented in order to produce an acceptable product. When good quality cream of low acidity is frozen, its flavor usually remains acceptable during storage. Trout and Scheid (1943) found that copper-free cream, pasteurized either at 165°F. (74°C.) for 15 min. or at 185°F. (85°C.) for 5 min., did not develop an oxidized flavor. However, when 1 p.p.m. of copper was added to the cream after pasteurization, oxidized flavor invariably developed during frozen storage. Homogenization of such cream was found to have only a very slight inhibitory effect on the copper-induced oxidized flavor. They concluded that when high-quality cream was produced and handled, free of copper contamination, and was adequately pasteurized, homogenization was not necessary to retard the development of oxidized flavor. Homogenization had a slightly beneficial effect, however, in retarding the development of copper-induced oxidation. Creams which were susceptible to the development of off-flavor usually showed such a defect during the first three months of frozen storage.

Only high-quality cream of low bacterial count and low acidity should be prepared for frozen storage. Several workers have observed that creams of high acidity (0.15% or higher) become unacceptable after 2 or 3 months, developing oxidized and other off-flavors. Sugar added to the extent of about 10% of the weight of the cream helps to maintain a satisfactory flavor.

Gelpi *et al.* (1952, 1955) studied the effects of adding recognized antioxidants to delay or prevent development of oxidized flavor in stored frozen cream. In the absence of copper, several antioxidants delayed development of oxidized flavor for at least six months. In the presence of added copper, only ethyl caffeate retarded off-flavor development beyond five months and only in summer cream; it was ineffective in winter cream. These results were obtained when the frozen cream was stored in standard glass milk bottles. When storage was in metal cans, the antioxidants were much less effective. Gelpi *et al.* (1955) found that ascorbic acid was not only ineffective in preventing the development of oxidized flavor but actually accelerated it. When ascorbic acid was combined with ethyl hydrocaffeate, with or without the presence of copper, the cream did not develop an oxidized flavor during twelve months' storage.

Body Changes in Frozen Cream.—When cream is frozen there is a tendency to disrupt the fat emulsion and to destabilize the milk protein. The physical equilibria of both components is changed, depending upon the severity of freezing conditions. This has been discussed in Chap. 10, Vol. 2.

Half or more of the fat in 50% cream can be destabilized and will “oil off” on thawing the cream to temperatures above the melting point of the fat. Rapid freezing tends to lessen the amount of fat de-emulsified as the water in the cream freezes. The fat destabilization will not be noticeable or objectionable unless the cream is thoroughly melted before it is added to ice cream mix or other food. Added sugar lowers the freezing point of cream and this protects the fat emulsion to the extent that ice formation in the cream is lessened. The fat of frozen cream can be completely reemulsified by homogenization either as cream or as the complete food product in which the cream is used.

Cream of 50% fat contains only 1.7% milk protein in contrast to 3.3% in fluid milk and 10% in a 3:1 milk concentrate. Plastic cream of 80% fat contains only 0.7% protein. The seriousness of the protein destabilization problem in frozen dairy products decreases with decrease in protein. If the protein in frozen cream becomes difficult to disperse on thawing because of prolonged storage or fluctuating storage temperatures, mild heat and stirring will usually disperse it.

Preparation of Frozen Cream.—Cream of 50–55% fat may be frozen and held in storage for future use by the following procedure (Heinemann 1967). Good quality cream (free of copper contamination) is pasteurized, cooled to 40°F. (4°C.) or lower and run into round plastic lined 2½- or 5-gal. containers for freezing. The containers are put into a cold room, such as an ice cream hardening room, where they are stacked to freeze and hold until they are to be used. When the containers are round they can be stacked close together, permitting air circulation between them. Freezing can also be done in plastic pouches which can be placed between refrigerated plates. More rapid freezing can be attained by slush freezing (without air incorporation) in a scraped-surface freezer (see preparation of plastic cream).

The National Research Development Corp. has been granted a British Patent (National Research Development Corp. 1964) covering a method of freezing cream. Unhomogenized cream is filled into an oxygen-impermeable container of good thermal conductivity. Six to ten per cent of the internal volume of the container is left unfilled. The containers are sealed hermetically. The cream is frozen by immersion in a liquid at approximately –38°F. (–39°C.) or by exposure to a blast of cold air or gas at –50°F. (–46°C.). The containers are stored at approximately 5°F.

($-15^{\circ}\text{C}.$). The frozen cream resulting has been stored for 12 months without deterioration, retaining the characteristics and whipping properties of the original cream when thawed.

At the time of use, frozen cream is removed from the freezer and held overnight to soften the surface in contact with the container. It is then easily removed. Where large quantities must be thawed quickly, the frozen cakes are passed through a suitable machine for breaking up and melting.

A German study (Storch 1965) gives total costs for an operation involving storage of up to 30,000 kg. (66,138 lb.) of cream annually as 35 D.M. / 100 kg. (\$4 / 100 lb.) of 45% fat cream.

Containers for Frozen Cream.—During the early years of freezing cream in the United States, 30- or 50-lb. metal cans were used as containers. Wire fastened to the two handles held the lids secure. The $2\frac{1}{2}$ - or 5-gal. single-service, round plastic-lined fiber ice cream container has replaced the tin can. The round shape permits air circulation even though the containers are stacked close together in the freezing room. The polyethylene liner is fastened to the cardboard stock of the container so that when the cream is partially thawed it drops out free of the liner. When a flexible or loose polyethylene bag is placed within a rigid container it is difficult to remove the cream completely, because part of the cream becomes enmeshed in the folds of the polyethylene liner, and the residual product cannot be easily or completely stripped out. Plastic coated rectangular boxes without other liners are also used as containers for cream. The filled containers occupy less space in storage than round cartons, but there must be provision for air circulation during freezing, or freezing must be done between plates.

Plastic bags (100 x 70 x 5 cm.) are used as containers for frozen cream in Europe (Krediet 1964). The bags permit the cream to be frozen in slabs between plates, and the slabs may be stacked on pallets for transportation and storage.

Thawing Frozen Cream.—Small quantities of frozen cream may be thawed by removing the bags, cartons, or cans from storage 1 or 2 days before use. The melted cream on the surface of the cake frees it from the container so that it can be dumped to mix with other liquid food. When quantities are large some mechanical aid must be employed for thawing. One U.S. company made an ice breaker into a frozen cream crusher by tinning the drum and making the bushings sanitary (Heinemann 1967).

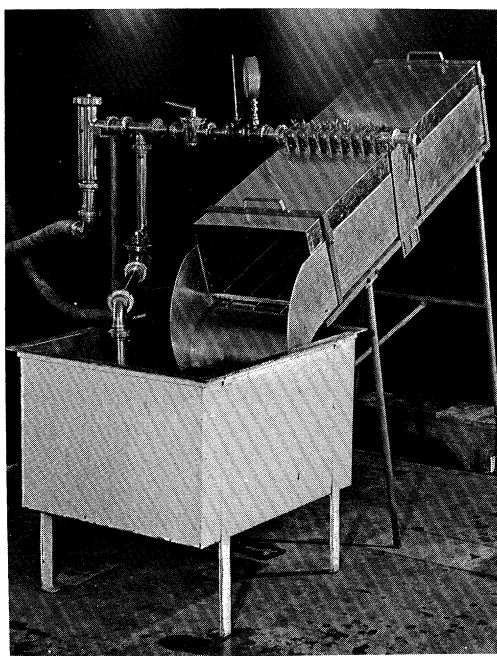
A German method for thawing frozen cream has been described (Jentjens 1962). Frozen blocks of cream are crushed and defrosted in a vat provided with a central vertical stirrer and a system of pipes coupled to a pasteurizer. The pieces of frozen cream are mixed with whole or skimmilk.

at a temperature not exceeding 77°F. (25°C.) and the mixture is warmed by passing milk at 77° to 86°F. (25° to 30°C.) through the pipe system.

A cream thawing unit developed at the dairy experiment station at Kiel under direction of Dr. Ing. G. Walzholz has been described (Gronau 1966). It is illustrated in Fig. 61 and 62. The cream to be defrosted should be frozen in slabs. The slabs are fed into the thawing unit (Fig. 61) which cuts them into thin slices (Fig. 62). The cutting is done by jets of warm milk as the slabs slide by gravity at a controlled rate down the chute. The slices of cream drop into a vessel in which the final defrosting takes place. The temperature of the nozzles is approximately 158°F. (70°C.); the height of the cream layer cannot exceed 30 cm.

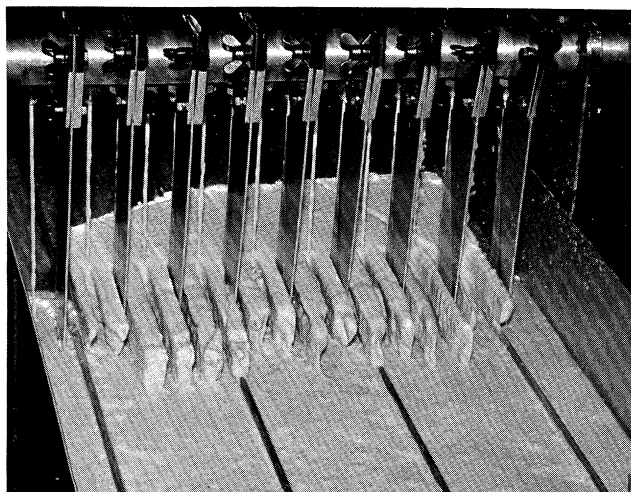
Plastic Cream

Based on a specific quantity of fat, plastic cream of 80% fat occupies only $\frac{5}{8}$ of the storage space of cream, requires fewer packages, and contains correspondingly less water to freeze. Cream of 50% fat contains about 45% water, whereas cream of 80% fat contains only about 18% water. Some



From Gronau 1966

Fig. 61. Pilot plant frozen cream thawing unit, Gronau/Rogge system



From Gronau 1966

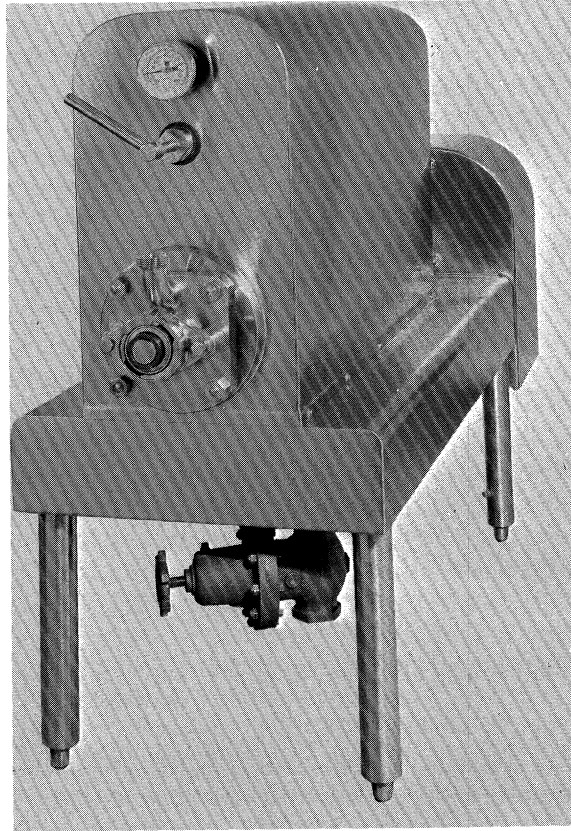
Fig. 62. Frozen cream thawing unit in operation, Gronau / Rogge system

Other machines for melting frozen cream and butter have been devised using, for example, internally heated rotating discs.

ice cream manufacturers consider that plastic cream produces better flavor and body in ice cream than does frozen cream. In spite of the advantages of plastic cream as a vehicle for storage of milkfat, Table 30 shows that 5 times more 50% cream is held in frozen storage than plastic cream. Two centrifugal separations are required to manufacture plastic cream, whereas one suffices for 50% cream. A further advantage of the lighter cream is its free-flowing property before freezing and after thawing. No special handling equipment is necessary.

Preparation and Storage of Plastic Cream.—Plastic cream is prepared by reseparation of 40% pasteurized cream to yield cream of 80% fat. The heavy product is chilled in a scraped-surface freezing unit or in a converted ice cream freezer, and is drawn directly into suitable containers such as are used for frozen 50% cream. The cream emerges from the chiller at about 40°F. (4°C.) or lower. Suitable slush freezing units are pictured in Fig. 63 and 65. Figure 64 is a pump suitable for use with the slush freezer shown in Fig. 63. The scraped surface continuous coolers of Fig. 63 and Fig. 65 are versatile units for cooling, slush freezing, or plasticizing cream and high-fat, high-viscosity dairy products.

The containers of chilled plastic cream are placed in a hardening room at 5°F. (−15°C.). Cream packaged in this way and held for two years has



Courtesy of CP Division, St. Regis Paper Co. (1967)

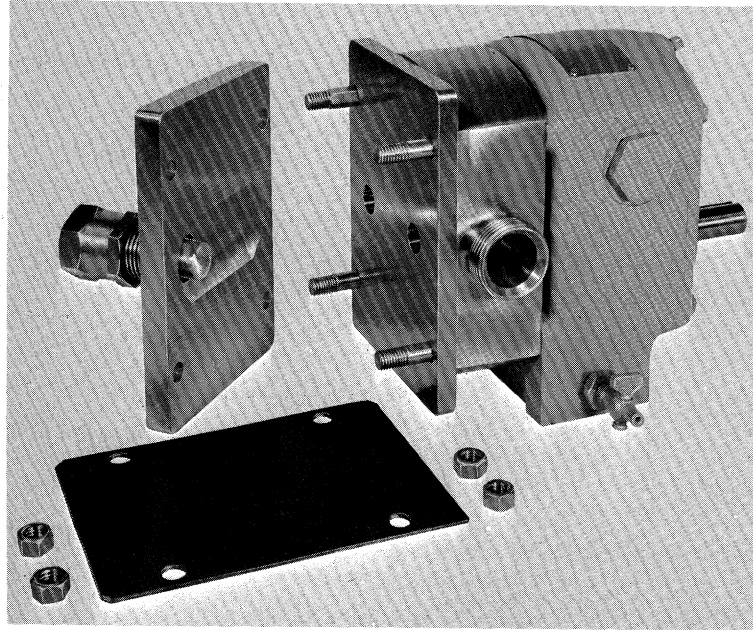
Fig. 63. Swept-surface heat exchanger

This scraped-surface type of heat exchanger is used to chill and slush-freeze milk products. It uses direct-expansion ammonia (or other refrigerant) as the chilling medium. The refrigerant accumulator, pressure regulator, and other controls are self-contained.

been reconstituted to a fluid milk of satisfactory flavor. Oxidized flavor was never a problem during a decade of production (Heinemann 1967).

Plastic cream has been used for the storage of milkfat by a Dutch butter factory (Anon. 1965). Forty per cent cream is received, neutralized, pasteurized, separated to 80% fat, frozen in molds at -40°F. (-40°C.), and the slabs are wrapped in plastic and stored at 10°F. (-12°C.). Frozen summer cream blended with fresh winter cream make excellent butter.

Plastic cream represents less of a thawing problem than does normal cream, since it contains less ice. It may be run through an ice breaker on



Courtesy of CP Division, St. Regis Paper Co. (1967)

Fig. 64. Stainless sanitary rotary pump with vented cover

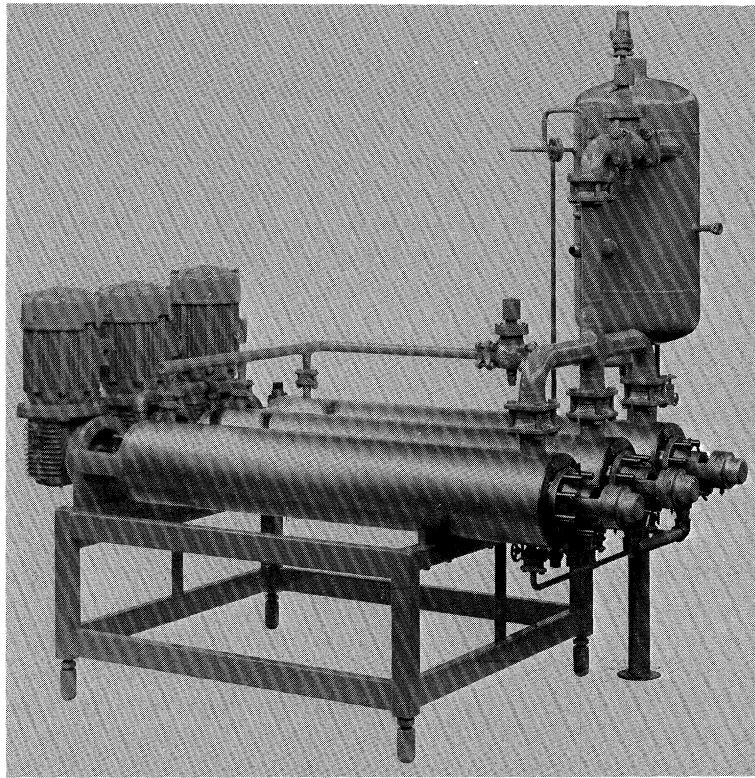
This rotary pump has 5-lobe rotors made of a resilient rubber-like material. The model shown is equipped with a vented cover which permits recirculation or relief at a pre-set discharge pressure. Pumps of this type used in conjunction with slush freezers prevent damage to downstream equipment from excessive pressure due to freeze-up or other line stoppage.

removal from storage or it may be allowed to remain out of refrigeration for about two days to soften, at which time it can be removed from the container and put through a crushing machine.

Frozen Whipping Cream

Neither frozen whipping cream nor frozen whipped cream is an article of commerce. Whipping cream should contain 30 to 35% fat. It should not be homogenized at a substantial pressure, because this would greatly reduce whipping properties. Freezing damages cream body, but addition of sugar before freezing, by decreasing ice formation, helps to produce a smooth bodied product in thawed cream. Any of the frozen creams can be re-emulsified in prepared foods to overcome defects in body which develop during freezing.

A procedure for preparing frozen whipping cream of 32% fat was developed by Storck (1961). Pasteurized cream was filled into flat plastic con-



Courtesy of Votator Division, Chemetron Corp. (1967)

Fig. 65. Scraped-surface chiller-freezer-crystallizer jacketed for direct-expansion ammonia or freon

Unit consists of a rotating bladed shaft within a jacketed insulated tube. Viscous products can be cooled from any temperature to slush-freeze if desired.

tainers or lined cartons and frozen rapidly in 30 min. at -4° to -22°F . (-20° to -30°C .). Fast freezing produced fine ice crystals and tended to avoid destabilization. The product was satisfactory in smell, taste, and serum separation, but volume increase on whipping was impaired and flocculation was noticeable.

Frozen whipped cream was prepared on a laboratory and commercial scale by Downs *et al.* (1960). Cream of 35% fat was pasteurized, and stabilizer, sugar, and vanilla flavoring were added. The cream was whipped, packaged in plastic bags, and frozen at -20°F . (-29°C .). After freezing and storage under various conditions examination showed there was body

and texture deterioration after three months. During the early storage period consumer acceptance was good.

COLD STORAGE OF BUTTER

Butter is the most popular form of product for storage of milkfat. It keeps well at low temperatures for periods of a year or more and it is less sensitive to temperature changes than many other dairy products. For many years after World War 2, the U.S. Government in its price support activities held large quantities of butter in storage. When the quality of the original butter was good and the storage temperature low and constant, the results were quite satisfactory.

Butter must be stored in the cold, but usually it is not considered as a product preserved by freezing. Butter will freeze at low storage temperatures but freezing has no effect on its characteristics that are noticeable after it has been thawed. Low-temperature storage of butter such as 0°F. (−18°C.) is advocated to prevent flavor deterioration and otherwise to keep it in a fresh, attractive condition.

Butteroil

Butteroil and anhydrous milkfat are terms used to designate pure milkfat that is substantially moisture-free (usually less than 0.2% water). Butteroil is prepared from butter, while anhydrous milkfat is made from cream. Both products may be held at room temperature without physical damage, whereas the body of butter is destroyed when the fat is melted. Butteroil and anhydrous milk fat gradually deteriorate in flavor when held at room temperature but they remain in good condition during 1 to 2 years of storage at 40°F. (4°C.) or lower. Containing no water, they do not freeze, but the fat components crystallize as storage temperatures are lowered.

Dried Butter

Dried butters of various compositions have been prepared on a limited scale in attempts to reduce the high storage costs associated with cold storage and rotating stocks of butter. These dried products contain at least 80% fat and varying quantities of nonfat milk solids. One new product appears to contain the normal butter ingredients less water. It has a minimum fat content of 97 to 98% and a water content of less than 0.1% (Strobel 1967). Dried butter has not yet attained commercial significance and is not a factor in affecting stocks of cold storage butter.

Freezing Characteristics of Butter

Unsalted butter freezes at 32°F. (0°C.), but most butter is salted. If it contains 2% salt, the water freezes at 15.8°F. (−9°C.); at 3.5% salt, its

water is frozen at -3.6°F. (-19.8°C.). The cryohydric point of sodium chloride is -4.2°F. (-21.2°C.). Below this temperature some salt crystallizes during frozen storage, but it readily dissolves when the butter is warmed. As butter is cooled there is a gradual formation of ice. The salt remains in the unfrozen portion until its concentration is such that it crystallizes. When butter is thawed there is a steady rise in temperature as the ice slowly melts and crystallized salt dissolves in it. The gradual freezing and thawing as temperature changes, which occurs in salted butter, avoid the destructive effects noted in products not provided with such a system buffered against freezing damage.

Both cooling and warming curves for salted butter are smooth and regular. They are affected to some extent by the fact that the specific heat of water is 1.0 while that of ice is only 0.5. Heat conductivity of butterfat is lower than that of water and air bubbles in butter contribute further to lowering its heat conductivity.

Cold Storage of Butter

The characteristics of butter under various freezing and thawing conditions may be important in relation to its commercial handling (McDowall 1953). Water expands on freezing, while butterfat shrinks, but these changes in volume apparently have not been correlated with changes in the physical properties of butter. When only a fraction of the water in salted butter is frozen, less refrigeration is required to lower it to a given temperature than would be required to freeze the water in unsalted butter at the same temperature. Repeated changes in the storage temperature of butter should be avoided, since the surface of the butter reaches air temperature more rapidly than the center of the block and frequent changes in temperature may therefore cause the surface butter to be inferior to that at the center.

Storage Temperatures.—Cold storage temperatures should be as low as possible, certainly no higher than -4°F. (-20°C.) if the holding time is to be several months. A preferred temperature is -20°F. (-29°C.) for long-term storage (1 yr. or more). If the butter is to be held for only 2 or 3 weeks, 40°F. (4°C.) may suffice. Pont and Gunnis (1958) found the temperature of storage to have no significant effect on the grade of butter after storage when storage temperatures were between -10°F. (-23°C.) and 12°F. (-11°C.). The grade loss in score was between 0.9 and 1.7 points after 6 months of such storage. Cracking in the stored butter resulted from excessive handling when butter was frozen.

Contact freezing of butter was found to produce excellent results (Malinowska 1965). The wrapped butter was passed through a multiple plate freezer, where it was frozen in single layers and where the time to reduce

the temperature of the butter to -22°F. (-30°C.) was 57 min., compared to 71 hr. in a tunnel freezer and longer than 120 hr. in a cold storage room with the butter in tubs. The plate-frozen butter kept significantly better than the butter frozen by traditional methods. The freezer could be made part of the production line.

Preparation of Butter for Storage.—The prestorage treatment of butter has an important effect upon its subsequent keeping quality. Butter that is placed in a freezer within 2 or 3 hr. of its manufacture will show markedly better keeping quality than that which may have been exposed to 40°F. (4°C.) for several days before freezing.

Butter for storage may be manufactured by either the churn or the continuous process. The cream is always pasteurized so that it is free of proteolytic types of organisms and contains largely only starter cultures for flavor production.

The butter should be wrapped in gas-tight foil or film impervious to light, placed in cartons or boxes and transported immediately to cold storage. A study of several combinations of wrapping materials showed that those with an aluminum foil base held butter in the best condition during storage (Pedersen and Fisker 1966). Storage temperatures should not fluctuate, and the boxes of butter should be stacked so that air can circulate around them.

Jasper (1962) recommends that butter be packaged directly from the churn into retail units which after storage at 0°F. (-18°C.) may be supplied to the retail shop while still in a frozen state. He placed 50-gm. packages of aluminum foil-wrapped butter between refrigerated plates at -22°F. (-30°C.) and noted they were frozen in 31 min., many hours sooner than large commercial blocks of butter. The small blocks of rapidly frozen butter kept better than that frozen in large blocks. The specific refrigeration requirement was 33.2 kcal./kg. butter, which is 27% less than for block butter.

Storage Changes in Butter

Butter is subject to deterioration in flavor and body during storage. Microbiological activity all but ceases at the lower temperatures and often actual numbers of organisms decrease but not without leaving residual effects on flavor.

Flavor in Storage Butter.—Butter is held in cold storage in a frozen condition to preserve its flavor, but butter is notorious for the rapidity with which it will absorb flavor from its environment. It is therefore important to keep storage rooms free of foreign odors and to wrap the butter in foil or other wrapping material which will not permit the penetration of air and off-odors.

If the butterfat has been unduly exposed to oxygen in the presence of copper, oxidized flavor will develop. Addition of 0.015 to 0.02% ascorbic acid has been found useful in slowing the rate of this copper-induced oxidation. Pont and Birtwistle (1966) have studied lipid oxidation in cold-stored butter in relation to its copper content and the pH of the serum. In butters with serum reactions between pH 6.6 and 8.0 the more alkaline butters had higher peroxide values and lower thiobarbituric acid values after storage.

The development of oxidized flavor in butter was found not to be significantly retarded by antioxidants during storage at either -18°F. (-28°C.) or 38°F. (3°C.) (El-Negoumy and Hammond 1960).

Antioxidants and synergists applied to the paper wrapper were claimed to afford substantial protection to butter, and its keeping quality was doubled as compared with controls. But there was rapid quality deterioration and spoilage (probably enzyme action) after 20 to 26 months of storage at 5°F. (-15°C.) (Zhedek *et al.* 1965).

Surface taints on 65-lb. cubes of butter were controlled by use of impervious cardboard laminates and by coating the inner liner of the carton with a moisture- and gas-tight material (Parodi 1966).

Bacteriological Changes in Butter in Storage.—Butter, under normal storage conditions, is a poor medium for bacterial growth. In general, the numbers of bacteria decrease during storage until there may be only 10% of the original population viable after 8 to 12 months' storage at 0°F. (-18°C.). However, butter containing large numbers of bacteria, especially lactic acid producers, has inferior keeping quality. In one test, control butter kept well for 6 months at -0.4°F. (-18°C.), while test samples inoculated with coliform bacteria were in poor condition after 3 months (Tso-nev 1964). Drabek (1964) studied the changes in total counts of proteolytic, lipolytic, and coliform bacteria, yeast and molds during storage of butter at 21°F. (-6°C.). The counts decreased with time and decreased more rapidly with high initial contamination. While all of the organisms showed a general decline in storage, the proteolytic bacteria were considerably inhibited by the presence of lactic acid but were not very sensitive to storage temperature.

Treatment of Butter After Storage

Butter that is removed from freezer storage should be placed in consumption channels at once. Bacteriological counts may have been lowered by storage treatment, but quality may have deteriorated. Undesirable physical changes sometimes occur. If a rapid return to higher temperatures is required, Sommer and Sommer (1965) suggest defrosting large blocks by contact with electrically heated copper bands.

The body of butter that has become coarse or uneven in distribution of moisture or fat can be reworked to produce a uniform, fine crystallized structure and improved spreadability. The plasticizing section of a continuous butter plant or the Votator chiller-freezer-crystallizer shown in Fig. 65 may be used to do this.

Printing Butter After Storage.—Butter should not be frozen before molding. Thomasos and Wood (1963, 1966) point out that the equipment used in printing butter (cutting and packaging) may cause the butter to show the presence of free moisture on its surface after the bulk-stored product has been tempered and subjected to the manipulation that takes place in the printing operation. When butter is removed from cold storage it is usually tempered in the range of 48° to 52°F. (9° to 11°C.) before printing. In examining the effect of four types of butter printers on the free moisture produced by the printing operation, differences were found in each printer. Printers employing extruders or rotating augers to force the butter into a mold caused moisture droplets to increase from less than 20 μ to a range of greater than 20 to 100 μ . Printers designed to handle soft butter out of the churn by forcing it with rotating polygonal rolls into the molding section did not change moisture distribution. The problem of free moisture and leaky butter resulting from the printing of cold storage butter could be eliminated by use of a homogenizing mechanism that would exert a high shearing stress.

FREEZING PRESERVATION OF MILK

Fresh fluid milk and milk concentrated to a ratio of 2:1, 3:1, or 4:1 have been frozen experimentally for preservation. Freezing of fluid milk and of a 3:1 concentrate have had limited commercial application.

Freezing of milk is satisfactorily accomplished by placing it in suitable retail containers in a cold storage room. However, attempts have been made to freeze milk rapidly by spraying it into a cold air stream at about -20°F. (-29°C.). The expense of such a procedure has thus far outweighed its advantages. A recent study examined the properties of milks quick-frozen at various temperatures (Coppens and Hote-Baudart 1965).

The effect of freezing on the bacterial flora of milk is considered under "Frozen Cultures" later in this chapter.

Fennema and Powrie (1964), in reviewing low-temperature food preservation, briefly discussed changes that were found to occur in milk and milk products during freezing and frozen storage. Winder (1962) has reviewed problems concerned with the physical-chemical stability of frozen milks. This is also discussed in Chap. 10, Vol. 2 of this work.

Freeze-Drying Milk

Although milk has been freeze-dried, this process is usually applied to pharmaceuticals, cultures, fruits, and fruit juices (see Chap. 14). Freeze-drying is more expensive to carry out than other methods of milk preservation. It is not practical to freeze large quantities of milk and then remove the ice by sublimation. The fat in freeze-dried milk is susceptible to oxidation just as is the fat in other milk products. The freeze-drying of milk has thus far not shown advantages which have encouraged its commercial development.

A process for continuous vacuum belt drying of milk has been developed which produces an excellent dried whole milk with good dispersibility and keeping quality when held at 40°F. (4°C.) (Schoppet *et al.* 1965). This is not a true freeze-dry process, however, since the water in the milk is not actually frozen before it is vaporized.

Freezing Fluid Milk

Fresh milk may be frozen to preserve it for shipment to inaccessible places or to hold it for future consumption. During World War 2 frozen homogenized milk was used to supply fresh milk to patients on hospital ships. This successful use aroused considerable interest in the product. The milk was acceptable when it was thawed after storage in a frozen condition for three months. After this period the quality of the product varied, although some reports indicated acceptability for as long as 6 to 9 months of storage. During the longer periods of storage two major problems developed—flavor deterioration and separation of milk solids upon thawing. Development of these defects can now be delayed by proper processing.

Processing Milk to Prevent Oxidized Flavor.—The mild, delicate flavor of fluid milk products can be retained in frozen storage better than by any other method of preservation. The stale flavor that inevitably develops in concentrated and dried milks is rare in frozen milks. But frozen milks are susceptible to the development of a typical oxidized flavor.

There are several approaches toward prevention of oxidized flavor in frozen milks. Bell (1948), Bell and Mucha (1949), Babcock *et al.* (1949), Anderson *et al.* (1950), Bell and Mucha (1951), and Babcock *et al.* (1952), studied the use of ascorbic acid as an antioxidant. In general, their collective findings indicated that ascorbic acid, while helpful, did not provide complete protection. Similarly, other workers have found that conventional antioxidants do have a mild repressing effect on appearance of oxidized flavor. Gelpi *et al.* (1962) tested various antioxidants and concluded that sodium gentisate was superior to the others. Milk containing 0.01% of this chemical could be held 6 months at -10°F. (-23°C.) without develop-

ment of oxidized flavor, but if copper was added the flavor appeared in 4 months.

Heating above pasteurization temperature (161°F., 71.7°C.) will retard development of oxidized flavor, but conditions for producing cooked flavor will be approached (Bell and Mucha 1951). Heating and homogenizing fluid milk is a simple and effective means of protecting it against oxidation. Oxidized flavor can then be retarded by a combination of measures: small additions of ascorbic acid; pasteurization at relatively high temperatures; homogenization; and finally, by being certain that the cows producing the milk receive adequate quantities of alpha-tocopherol (1 gm.) in their daily rations (King *et al.* 1966).

The volatile sulfhydryl compounds developed in the milk by heat, which combat oxidized flavor, are dissipated in frozen storage after 1 to 2 weeks. When they are present in the milk the cooked flavor is pronounced, but this gradually subsides so that there is an actual improvement in flavor during the early storage period.

Processing Milk for Physical Stability.—Separation of milk solids during thawing of frozen milk can be delayed by addition of a small quantity of citric acid after pasteurization and by homogenization, procedures which also retard development of oxidized flavor. The sodium polyphosphates prevent casein insolubility by their peptizing action but they are usually not needed to stabilize fluid milk.

Homogenization alone is effective in protecting the fat emulsion of milk so that free fat will not oil-off on the surface of the thawed product.

Preparation of Milk for Freezing.—Fresh Grade A milk of low bacterial count and with a fat content of about 3.8% should be pasteurized, preferably by holding at 155°F. (68.3°C.) for 30 min., and packaged in half-pint, pint, or quart paper containers. The common polyethylene-lined paper milk containers provide excellent packages for frozen milk. The milk should be homogenized at about 135°F. (57°C.), as it is raised to pasteurization temperature. The homogenization pressure should be at least 1,500 p.s.i. Use of the following additives after pasteurization will essentially double the storage life of the frozen milk. For each liter (quart) of milk add 2 gm. of sodium citrate and $\frac{1}{10}$ gm. of ascorbic acid. Babcock *et al.* (1949) found that of a number of chemicals that stabilized both flavor and sedimentation of the milk, these were the most effective. After the stabilizers have been added, the milk should be packaged in paper cartons, using conventional equipment.

Freezing and Storage of Milk.—Babcock froze and held his samples of milk in wax-lined cartons at 0°F. (−18°C.). Higher freezing temperatures are undesirable but lower temperatures may improve quality. Separation is not noticeable in the thawed milk until after it has been stored 150 days,

and flavor is usually normal up to about 120 days, after which a slight oxidized flavor may develop.

Recently, Radema and Dijk (1965) reported that pasteurized milk (4% fat) frozen and stored at 14°F. (−10°C.) became destabilized in 40 days; at −4°F. (−20°C.) there was impaired stability in 6 to 11 months; at −22°F. (−30°C.) stability was retained for more than 11 months. Homogenization before freezing had a slight adverse effect on protein stability, but it prevented the formation of a surface layer of melted fat on the thawed milk.

It can be concluded that pasteurized homogenized milk frozen and held at 0°F. (−18°C.) can be shipped to almost any point on earth and should arrive in acceptable condition. Higher storage temperatures may be used, but temperatures above 10°F. (−12°C.) are not recommended, because the storage life of the milk will be appreciably shorter.

Frozen Concentrated Milk

Milk can be concentrated in a vacuum without impairing its flavor, but preservation of the concentrate during long periods of storage has been a challenging problem. Reduction of the sterilized or cooked flavor of evaporated milk by substitution of high-temperature, short-time sterilization for the long-hold process has been effective in improving its quality. But its flavor is still more cooked than is acceptable for most beverage purposes, and room-temperature storage brings staling changes. Frozen concentrated milk more nearly meets the rigid quality requirements of the American public, but extensive use of this product has been delayed pending solution of technical and economic problems.

The same defects occur during the storage of frozen concentrated milk that occur in frozen pasteurized milk, but fat separation is less in the concentrate because the higher content of milk solids protects the fat emulsion and because the milk has been homogenized. Similarly, oxidized flavor in the concentrate can usually be avoided by heating the milk to a relatively high pasteurization temperature and homogenizing it before concentration. Thickening increases in magnitude as the concentration of the milk to be frozen is increased.

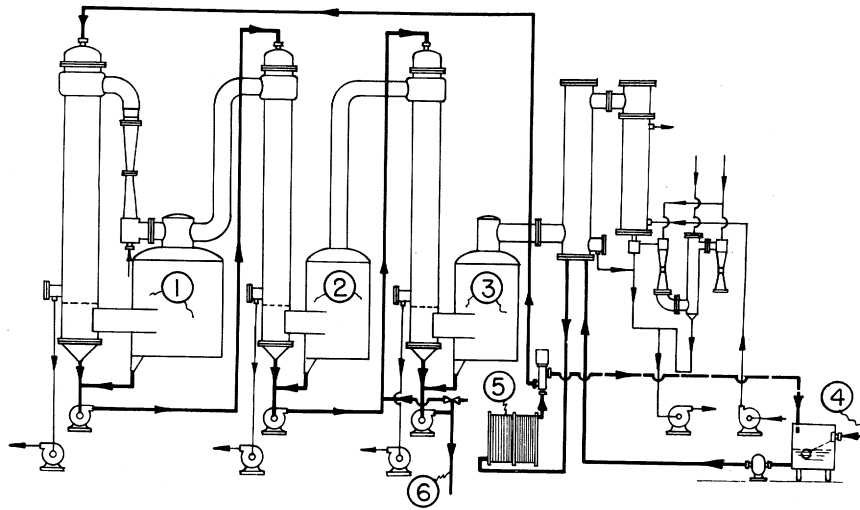
Preparation and Freezing of Concentrated Milk.—An early process patent describes a very simple procedure for preserving concentrated milk by freezing (Webb 1934). Fresh milk was pasteurized, condensed under vacuum to $\frac{1}{3}$ its original volume, cooled, sealed in cans, and frozen at 10°F. (−12°C.). The product was satisfactory after storage at this temperature for 2 or 3 weeks, but after that time the thawed reconstituted product showed fat separation and protein coagulation which made it unsuitable for commercial production. Research has shown how the undesirable change that occurs in frozen concentrated milk can be greatly delayed. This work has

been discussed in Vol. II, Chap. 10. Acceptable processing procedures will be discussed here.

One of the advantages of freezing concentrated milk rather than fluid milk is that there is an attractive saving in container and shipping costs. Evaporated milk at a 2:1 concentration has always been popular, but it is very difficult to manufacture a sterilized milk at 35% solids, because at this concentration the milk protein lacks the heat stability necessary if the product is to be sterilized. When the concentrate is frozen, so that no sterilization is necessary, a 3:1 concentration becomes practical.

Concentration of Milk Under Vacuum.—Milk is concentrated under vacuum to reduce weight and volume, saving container, storage, and shipping costs. For food manufacture, as in production of bakery products, ice cream, and confections either a concentrate or a powder is needed.

The concentration of milk as practiced in a large plant by means of a triple-effect falling-film evaporator is a very efficient operation. A diagrammatic outline of the process drawn by Hanrahan (1967) is shown in Fig. 66. Milk enters the first stage tube chest, descends as a boiling film, and enters the flash chamber where milk and vapor are separated. The vapor is used



From Hanrahan (1967). Information also furnished by A. W. Baumann, Arthur Harris and Co. Chicago, Ill.

Fig. 66. Triple-effect falling-film evaporator with vapor compression and heaters, for concentration of milk

(1) First stage, with operating temperature of 155°F. (68°C.); (2) second stage, with operating temperature of 140°F. (60°C.); (3) third stage, with operating temperature of 115°F. (46°C.); (4) flow of cold raw milk enters system, to be forewarmed by outgoing vapors from stage 3; (5) plate heater for pasteurizing milk; and (6) flow of condensed pasteurized milk leaving system.

to heat the tube chest of the next stage, while the milk is further concentrated in that chest. This is repeated in the third stage at a still lower temperature. Vacuum concentration of milk removes off-flavors, and since the temperatures used are no higher than pasteurization, no objectionable cooked flavor is added.

Freeze-Concentration of Milk.—Processes have been devised whereby milk, whey, and other liquids are concentrated by freezing part of their water content and removing the ice crystals (see also Chap. 4 of this volume). Concentration by freezing should be an efficient operation because the latent heat of fusion of water is 143 B.t.u. per lb., whereas its latent of heat of vaporization is 971 B.t.u. However, the cost of refrigeration and the mechanical difficulties attendant upon removing frozen ice crystals from milk or other dairy products have thus far retarded extensive commercial adaptation of systems of concentration by freezing. Energy costs might be greatly reduced in the concentration of such liquids as skim milk or whey if they could be exposed to subfreezing temperatures during suitable weather, and then concentrated by removal of ice crystals. This would provide small milk and cheese factories with a cheap means of removing water from their liquid products, but uncertain weather would jeopardize the usefulness of any process for concentration by natural freezing.

A number of freeze-concentration procedures have been reviewed by Whittier and Webb (1950). One method provides for the controlled growth of ice crystals and their removal in such a way as to minimize contamination of the crystals by solids of the fluid being concentrated. In another procedure, whey is concentrated to 30% solids, with a loss of 10% in the ice-crystal sludge. One inventor advocates the use of a large stainless steel cylinder, surrounded by brine, within which is mounted a screw conveyor. Scraping blades on the conveyor remove ice from the cylinder walls and carry it to a centrifuge that separates the ice from any milk that may have been carried over from the concentrator. In another process milk is frozen in the form of a soft block which fits into the basket of a centrifuge. The concentrate is spun off, the ice is removed from the centrifuge, and another block of frozen milk is placed in the centrifuge.

An abstract of a process developed by two Russian workers for the concentration of cheese whey by freezing was published recently (Tylkin and Kozlov 1964). Whey containing 5.2% solids was frozen at 25°F. (−4°C.), filtered, and pressed to separate the liquid phase from the ice crystals; these were subsequently washed to obtain a liquid with 11.8% total solids. The freezing operation was repeated several times in order to obtain 25 to 30% whey solids. A process for simultaneously concentrating and freezing milk has been described by Vidal *et al.* (1963). The milk is pasteurized, homogenized, and atomized so that the droplets fall through an ascending

cold air stream at -22°F . (-30°C .), while they are subjected to a vibratory motion. Obviously, both these concentration procedures would be cumbersome compared to modern methods of vacuum evaporation.

Smith (1964) patented a process in which milk was subjected to a nitrogen-stripping operation and then concentrated by freezing in absence of oxygen. The keeping quality of the N_2 -processed concentrate was superior to that of air-processed milk.

Lactose Crystallization and Gel Formation in Frozen Concentrated Milk.—Coagulation is retarded by removal of some of the lactose or by suppressing its crystallization (see Chap. 10, Vol. 2).

It is known that crystallization of the lactose in frozen concentrated milk appears to initiate protein flocculation. Only circumstantial evidence links the crystallization of lactose to the subsequent coagulation of milk protein during frozen storage. The relationship between the two observed changes is not well understood. Protein destabilization can usually be delayed for as long as lactose can be prevented from crystallizing in the concentrate. Seeding the concentrate brings rapid destabilization, but heating the concentrate or taking other precautions to avoid nuclei formation delays protein coagulation. The addition of freezing point depressants delays destabilization. Salt and sugar are effective in this respect.

Frozen Concentrated Milk Processes.—Six processes for manufacturing frozen concentrated milk will be described, but the last two are not commercially practical under present operating conditions. The processes described avoid three defects characteristic of early frozen concentrated milk products. These are coagulation of the milk protein during freezing, separation of fat, and development of an oxidized flavor.

Fat separation is retarded by increasing the solids content of the milk over that of normal milk and by homogenization. Oxidized flavor is retarded by homogenization and may be further delayed by use of antioxidants. The copper content of milk, when high, catalyzes the development of oxidized flavor and it is desirable to handle the milk under conditions such that copper contamination will not be possible. Stainless steel equipment should be used throughout for processing the milk. Milks of high natural copper and low tocopherol contents show an increased tendency toward development of oxidized flavor.

Enzymatic Hydrolysis of Lactose by Lactase.—This process was developed by Stimpson (1954) and Tumerman *et al.* (1954). To carry out the process, a supply of the enzyme lactase is necessary. This may be obtained commercially or it can be produced as a by-product during the growth of *Saccharomyces fragilis*. Much work has been done by Stimpson and associates on the production and purification of a flavor-free lactase of high activity. To greatly prolong the storage life of frozen concentrated milk, it is

necessary to hydrolyze only 10 to 15% of the lactose in the product. This is most easily done by separating 15 to 20% of the milk and hydrolyzing the lactose in the skimmilk fraction. The amount of enzyme preparation to use depends upon its potency and the quantity of lactose to be hydrolyzed. In general, 1.5 to 3% of enzyme by weight of lactose will hydrolyze 80 to 95% of the lactose. The enzyme is added to the skimmilk at 130°F. (54°C.) and the milk is held until about 90% of the lactose is hydrolyzed. Sufficient enzyme is used so that the hydrolysis is completed in about four hours. The enzyme is inactivated by pasteurization of the hydrolyzed skimmilk, which is then added to the raw whole milk. The cream previously removed from the hydrolyzed skimmilk fraction is also added back to the batch. The fluid whole milk, which now contains less than 90% of its original lactose, is pasteurized, preferably at 162°F. (72.2°C.) for 15 sec., homogenized at 1,500 p.s.i. or more pressure, concentrated under vacuum to 35% solids, cooled, packaged, preferably in metal cans, and frozen as rapidly as possible, usually in a cold room at about -20°F. (-29°C.). The enzymatic hydrolysis procedure more than doubles the storage life of the milk, which now reconstitutes to a smooth liquid product. Homogenization prevents fat separation, and together with concentration it usually retards the development of oxidized flavor. The concentrate is more resistant to oxidation than is the fluid milk.

Stabilization of Frozen Concentrated Milk by Polyphosphates.—Polyphosphates added to concentrated milk before freezing have been shown to stabilize it during frozen storage (Leviton *et al.* 1962, 1966). The most suitable form of polyphosphate is a tetra-polyphosphate glass in the form of a straight-chain product having an average number of phosphorus atoms of about 4.8 per chain. Products with longer chain lengths have been used and are also acceptable. The cyclic polyphosphates should be avoided. The tetra-polyphosphate glass is available commercially and is made by fusion of mixtures of mono-basic and di-basic sodium phosphates. The milk should be pasteurized at 162°F. (72.2°C.) for 15 sec., and concentrated to 35+ % solids. By slightly overcondensing, the polyphosphate may be added dissolved in water when the milk is standardized to the desired solids content. The amount of polyphosphate to use may range from 0.5 to 1.5 lb. per 100 lb. of milk solids, depending upon the degree of stability desired. The storage life of a 35%-solids milk concentrate can be greatly extended by adding about 1.0 lb. of stabilizer per 100 lb. of milk solids. For example, Leviton *et al.* (1966) found that milk held at 10°F. (-12°C.) began to show sediment after three days' storage and this increased progressively during a storage period of 90 days, whereas test samples containing 0.8 lb. of polyphosphate per 100 lb. milk solids showed no sediment during the 90-day

experimental period. Polyphosphates increase storage life of frozen milk even when the milk is seeded with lactose before freezing.

Use of Soluble Additives to Suppress Lactose Crystallization.—Sugar at a 5 to 10% level (based on the weight of the milk) may be used to retard lactose crystallization. Sweetening of the milk to this extent is unacceptable for many beverage uses. Of various other solutes of low molecular weight, Tumerman and Guth (1965) found that salts of alkali and alkaline earth metals were particularly effective. Sodium chloride is used in the following procedure taken from the work of Tumerman and Guth. The milk is pasteurized, concentrated under vacuum to 35% solids, of which the lactose content is approximately 13.3%. The milk is treated by dissolving sodium chloride in it at the rate of 0.25% by weight of the concentrated milk. After the salt has been dissolved, the milk is homogenized and again pasteurized and cooled. The treated milk is packaged in cartons or other suitable containers and placed in storage at 15°F. (−9°C.) or lower. After 20 weeks the concentrated milk, when thawed, should be of high quality and there should be no visible gel formation. A comparable batch of milk having the same solids content, but without added salt, and stored under the same conditions will show protein coagulation after six weeks of storage.

At this level of additive (0.25% NaCl), the flavor imparted to the milk on reconstitution to normal strength is scarcely noticeable. The effect of salt on flavor can be entirely overcome by adding flavoring materials such as chocolate or fruit flavors.

Processing to Retard Lactose Crystallization.—This process developed by Braatz (1961) and Braatz and Winder (1959) has been commercially successful in limited production and distribution tests. With minor variations the process is essentially as follows: the raw milk is pasteurized at a temperature not exceeding 165°F. (74°C.) for 16 sec., homogenized at 2,500 p.s.i., condensed to 36% total solids at a temperature below 140°F. (60°C.), then packaged in cans. The canned product is heated at 155°F. (68°C.) for 25 min., cooled without agitation, and frozen under quiescent conditions at 0°F. (−18°C.). The most important step in the process is the postcondensing heat treatment of the concentrate. This dissolves the lactose nuclei which may have formed in the milk during condensing or canning. Any agitation after cooling below 100°F. (38°C.) or during the freezing of the concentrate reduces the beneficial effects produced by the postcondensing heat treatment. Frozen concentrated milk produced under these conditions remains acceptable in flavor and body for about four months' storage at 10°F. (−12°C.). Success of the method is dependent both upon dissolving lactose nuclei which may have formed and upon using extreme care that nuclei do not reform during handling before the milk is finally frozen.

Fluctuations in storage temperature can be expected to trigger lactose crystallization and shorten the life of the milk.

Dialysis of Milk to Remove Lactose and Calcium.—This process was suggested by El-Negoumy and Boyd (1965) and in its present state of development is intended only as a laboratory procedure. About 50% of the lactose is removed by dialysis against a simulated milk ultrafiltrate of average composition, except that it is devoid of lactose. An alternate procedure is to partially remove the soluble calcium by dialysis against an ultrafiltrate devoid of calcium. The reader is referred to the original work for details of the dialysis procedure. After dialysis, the milk is forewarmed to 150°F. (66°C.) and vacuum-condensed to a concentration of 3:1. The concentrate is cooled quiescently to about 45°F. (7°C.) and then packed in plastic bags or other suitable types of containers. The milk is frozen at -15°F. (-26°C.) and held at this or at higher temperatures. When held at 15°F. (-9°C.) the milk should be stable for 30 weeks.

Crystallization of the Lactose and Its Removal from the Concentrate.—There is no present means to carry out this process on a commercial scale because of the high viscosity developed in the concentrate during lactose crystallization and the difficulty of removing the lactose crystals. Lactose can be removed from concentrated milk if sucrose is added to the milk before condensing (Webb and Williams 1934). The sucrose has a diluting effect so that the concentrate remains thin during the period of several hours necessary to obtain lactose crystallization. In the absence of sucrose, 3:1 whole milk thickens in a matter of hours, during the time required to crystallize the lactose. The most practical way to remove the lactose is by centrifuging the concentrate, but sometimes the milk reaches a gel-like consistency before a substantial quantity of lactose has crystallized.

Thawing Frozen Milk

Milk and concentrated milk are usually frozen in retail cartons not exceeding the 2-qt. size. On removal from frozen storage it may be thawed by immersing the container in warm water. If more thawing time is available holding overnight in a refrigerator is a satisfactory method.

If flocculated casein, lumps, or a gel is apparent on thawing, the protein can often be redispersed by warming and stirring the milk. Gelation is reversible to a certain point, but eventually, as storage is prolonged, irreversible coagulation occurs.

PRESERVATION OF CHEESE BY FREEZING

The freezing of cheese is usually avoided because of a tendency toward physical breakdown in body and structural characteristics caused by ice crystal formation. The freezing points and moisture contents of several vari-

eties of cheese are shown in Table 31, Chap. 10, Vol. 2. Salt added to cheese during making, and the soluble constituents developed during ripening, lower the freezing points of most cheese. Unripened, high-moisture cheese such as cottage cheese, which is very perishable, has a freezing point of 29.8°F. (−1.2°C.). Frozen storage is useful in preserving cottage curd, but deleterious physical changes must be avoided or overcome.

Freezing Ripened Cheese

Robertson (1966) has reviewed recent progress in Cheddar cheesemaking. There is no sign that the changes in this process, now developing from

Table 31
Storage Life of Dairy Products

Product (Commercial Pack)	Approximate Storage Life at Specific Temperatures			Critical or Dangerous Storage Conditions
	Months	Temperature °F.	Temperature °C.	
Butter (in bulk)	1	40	4	Above 50°F. (10°C.) or damp or wet storage
	12	−10	−23	
Butteroil (sealed, full tins; maximum moisture 0.3%)	3	70	21	Above 75°F. (24°C.)
	6	50	10	
Ghee (sealed, full tins)	9	32	0	Above 90°F. (32°C.)
	6	90	32	
	9	70	21	
	18	40	4	
Cream (50% fat)	12	−10	−23	Above 20°F. (−7°C.)
Plastic cream (80% fat)	12	−10	−23	Above 20°F. (−7°C.)
Frozen milk	3	−10	−23	Above 10°F. (−12°C.)
Frozen concentrated milk	6	−10	−23	Above 10°F. (−12°C.)
Frozen cultures	6	−10	−23	Above 10°F. (−12°C.)
Nonfat dry milk, Extra Grade (in moisture- proof pack)	6	90	32	Above 110°F. (43°C.)
	16	70	21	
Dry whole milk, Extra Grade (gas pack; max- imum oxygen 2%)	24	40	4	Above 100°F. (38°C.)
	3	90	32	
	9	70	21	
Sweetened condensed milk	18	40	4	Above 100°F. (38°C.) or below 20°F. (−7°C.), or dampness suf- ficient to cause can rusting
	3	90	32	
	9	70	21	
Grated cheese (in moisture-proof pack)	15	40	4	Above 70°F. (21°C.) or above 17% moisture in the product
	3	70	21	
Cheddar cheese	12	40	4	Above 60°F. (16°C.) or below 30°F. (−1°C.)
	6	40	4	
Processed cheese	18	34	1	Above 90°F. (32°C.) or below 30°F. (−1°C.)
	3	70	21	
	12	40	4	
Sterilized whole milk	4	70	21	Above 90°F. (32°C.) or below 30°F. (−1°C.)
	12	40	4	
Evaporated milk	1	90	32	Above 90°F. (32°C.) or below 30°F. (−1°C.) or dampness suf- ficient to cause can rusting
	12	70	21	
	24	40	4	

Source: Adapted in part from a chart compiled by Foreign Agr. Serv., Dairy and Poultry Div. (1966) from material supplied by Standardization Branch, Consumer Marketing Serv. and Eastern Utiliz. Res. Div. Agr. Res. Serv., U.S. Dept. Agr., Washington, D.C.

an ancient art to a fast, mechanized operation, will affect the deleterious changes caused by freezing.

Ripened cheese (Cheddar, Swiss) keeps well in cold storage above its freezing point, where microbiological changes slowly break down the cheese protein and fat to produce the mellow body desired in a well-ripened cheese. Excessive breakdown may occur if the ripening process is continued for too long a period of time. Ripening can thus be retarded if not altogether stopped by freezing the cheese. When this is done the smooth texture of the well-ripened cheese may become rough, mealy, and crumbly after freezing and thawing. There may be some recovery of body if ageing and protein breakdown is allowed to continue. The flavor of ripened cheese is not affected significantly by freezing and thawing. A reducing atmosphere is developed during ripening which protects the fat against oxidation.

Several years ago, the University of Minnesota received numerous requests for information from locker patrons and others concerned with the problem of what to do with excess cheese. To answer such questions, Morris and Combs (1955) studied the freezing of ripened cheese and stated that one can freeze and store cheese if the temperature of the locker is 0°F. (−18°C.) or lower, and if the cheese is tightly wrapped in foil to prevent moisture loss. But the body and texture of ripened cheese sometimes become undesirable as a result of freezing and thawing. Water tends to separate from the protein; this produces a crumbly and mealy body and a general roughening of texture. In some cases, excess whey gives a wet appearance to the curd particles.

Ripened cheeses that are to be frozen should be cut and wrapped in moisture-tight foil or film so that the packages do not contain more than about one pound. Freezing should be done as rapidly as possible, preferably at a temperature of −10°F. (−23°C.) or lower. The cheese should be thawed slowly in a refrigerator set at a temperature above its freezing point. Morris and Combs (1955) state that some cheese can be frozen and stored for at least six months. The varieties they mention are Cheddar, brick, Port du Salut, Swiss, Provolone, Mozzarella, Liederkranz, Camembert, Parmesan, and Romano.

Chemical Changes in Frozen Ripened Cheese.—Chemical changes were found to occur in ripened cheeses during storage at −10°F. (−23°C.) (Harper and Kristoffersen 1958). While there were only negligible changes in the free amino and free fatty acid contents of Romano, Provolone, Swiss, and Cheddar cheeses, there were appreciable losses of certain acidic carbonyl compounds. The alpha-acetolactic acid had completely disappeared from 2 of 3 Cheddar cheeses stored at −10°F. (−23°C.) for 1 year, and only a trace remained in the third. Oxalacetic acid had disappeared from

all three cheeses, but the other keto acid constituents were unchanged. There was no change in the concentration of the keto acids in Romano cheese stored for 1 month, but after 2 months oxalacetic acid was missing and neither oxalacetic, acetoacetic, or alpha-acetolactic acid could be detected after 3 months. As a result of this study the authors recommend caution in the interpretation of results of analyses of cheese placed in cold storage. Other workers have found increases in soluble nitrogen during storage of cheese at low temperatures, but in many cases the temperatures used, while being below the freezing point of water, were not below the freezing point of the aqueous phase of the cheese.

Freezing Fresh Curd Cheese

Fresh curd cheese, particularly the cottage cheese of the United States and the "quarg" of Europe, are high in water content and do not freeze well in their usual merchandizable form. In 1966, production of cottage cheese curd in the United States was 625 million pounds, while production of creamed cottage cheese was 836 million pounds (Statistical Reporting Service 1967). The storage life of cottage cheese in normal refrigerators is about 15 days, but production tends to be seasonal with milk production. A simple method of freezing fresh curd to yield a satisfactory product is still not available. If the high-moisture cheeses are to be preserved by freezing, their composition and method of handling during manufacture may be altered to reduce freezing damage.

Oxidized flavor may develop in frozen high-moisture cheeses containing considerable quantities of fat. When cottage cheese is prepared for frozen storage, usually only the curd without fat is frozen. Thus, the development of oxidized flavor in frozen cottage cheese curd may be avoided.

In a review of recent research on the manufacture of cottage cheese, Emmons (1963) discussed methods of storing the curd. When it was necessary or desirable to store cottage cheese curd, freezing was one of the best methods to preserve it. To overcome the weak and mealy condition of the thawed curd, salt was used to lower the freezing point of the water in the curd. The curd was salted lightly and stored in air-tight 50-lb. cans at 0°F. (−18°C.) or lower. Thawing was critical and should be done slowly over a period of 2 to 4 days. Rishoi *et al.* (1959) advocate holding curd that is to be frozen for at least 18 hr. before creaming it. They found that cottage cheese stored for 9 months at 0° to −10°F. (−18° to −23°C.) was only slightly less acceptable than freshly-made cheese. A number of investigators have immersed cottage cheese curd in brine; this prevented the cheese from freezing, but stopped bacterial deterioration. Emmons (1963) cites workers who found that curd could be held in 3% brine for 14 days, in 4% brine for 1 month at 45°F. (7°C.), or 2 months at 35°F. (2°C.), or in 6%

brine at 40°F. (4°C.) for 5 months or more. Curd preserved in brine should be packaged in plastic-lined containers, preferably in the absence of air.

Quarg is the fresh curd product made in Central and Eastern Europe; it resembles uncreamed cottage cheese curd. Russian workers (Bogdanova and Senkevich 1965) report tests on frozen quarg packaged in polyethylene film and other materials. The curd was preserved successfully by freezing it rapidly at a temperature below -18°F. (-28°C.), with subsequent storage below 0°F. (-18°C.). Krcal (1965), working in Czechoslovakia, found that quarg could be stored for 6 months without significant deterioration of quality if it was held at 0°F. (-18°C.). In all cases storage caused increases in total solids, depending upon the packaging material and the storage temperature. There was further loss of moisture during defrosting. No change was found in titratable acidity, but soluble nitrogen increased, particularly when the storage temperature was as high as 32°F. (0°C.). All quarg samples decreased in bacteria count during storage.

Preparation of Fresh Curd for Freezing.—Cottage cheese or the East European quarg may be prepared for freezing preservation. Cottage cheese should be made from clean-flavored skimmilk of low bacterial content which has been pasteurized at 161°F. (71.7°C.) for 15 sec., or at 145°F. (62.8°C.) for 30 min., or by some equivalent pasteurization process. A clean, active *Strep. lactis* starter culture should be used. At the time of cutting, the curd should have a titratable acidity of 0.48 to 0.50%. Either the "long-setting" or the "short-setting" process is satisfactory. Dry uncreamed curd of not more than 80% moisture is best for freezing. Yeasts, molds, and coliforms should be less than ten per gram. When prepared according to the above specifications, the flavor of the cheese should be stable for 6 months if held at 0°F. (-18°C.). The curd should be salted lightly, placed in suitable moisture-tight metal or fiber containers, and fast frozen in a freezer or between plates, preferably at -20°F. (-29°C.) or lower. After the curd is completely frozen the storage temperature may be raised, but it should remain between 0°F. (-18°C.) and -10°F. (-23°C.).

Freeze-Drying Cheese

A method for freeze-drying quarg (cottage cheese curd) has been reported (Evstrat'eva *et al.* 1960). The curd is placed under high vacuum on shelves cooled to -29°F. (-34°C.) and the moisture is reduced to about 3%. Plant output was 74.2 kg./day. The shelf-life of several ripened cheeses and of creamed cottage cheese has been increased up to one year by freeze-drying and inert gas packaging (Meyer and Jokay 1959; Jokay and Meyer 1959). Schulz (1966) found that a desirable porous structure could be obtained in freeze-dried cheese only when the moisture content in the nonfat portion of the undried cheese was 70 to 80%.

FREEZING STARTER CULTURES

The use of frozen cultures of microorganisms for manufacture of cheese and cultured dairy products is a recent industrial development. It eliminates some of the uncertainties and expense involved in daily transfers, and provides a ready supply of active starters for dairy processing activities. Survival of bacteria is excellent when cultures are frozen and held in a deep frozen state until they are thawed for use. Survival of up to 75 to 100% of the cells may be expected under optimum conditions.

Preparation of Cultures for Freezing

Frozen cultures are prepared by selection and growth of the organisms on suitable media to get them quickly into the late logarithmic growth phase. The growth media must be compounded not only to meet the growth requirement of the organism, but also to permit it to have a full complement of required enzyme systems for subsequent activities.

Many investigators have been concerned with the composition of the growth media, among them Duggan *et al.* (1959), Lamprech and Foster (1963), and Cowman and Speck (1965). Glucose tryptone broth, "deionized" dried whey, casein digest, and yeast extracts are among media components. Simmons and Graham (1959) found that the most active cultures were obtained when they were grown in skimmilk fortified with 2% added milk solids. Anderson (1962) inoculated single-strain isolates into "freshly pasteurized antibiotic-free skimmilk" and froze them in sterile polyethylene bags at 8°F. (−13°C.). Activity of his cultures was "not substantially diminished" by frozen storage for periods up to 12 months.

Kawashima and Maeno (1964) froze suspensions of *L. bulgaricus* and *S. lactis* in 10% solids reconstituted skimmilk at −29°F. (−32.6°C.) for 6 months and found acid production of the thawed and incubated cultures to be 35 and 68% of subcultured unfrozen cultures. They found that L-glutamic acid added to the medium before freezing stimulated growth after freezing and thawing.

Moss and Speck (1962) observed that injury and death due to freezing were more pronounced when cells were frozen in water than in skimmilk.

Johns (1956) noted that different starters varied in their reaction to frozen storage, but in every case there was marked improvement in survival of cells if the starter was neutralized to 0.16% acidity before freezing.

Temperature and Time of Freezing

Cultures have usually been frozen and held at −4°F. (−20°C.). Gibson *et al.* (1965) examined 16 single strain cultures for their ability to withstand freezing and storage at 0°F. (−18°C) and −10°F. (−23°C.). The strains used were *S. cremoris*, *S. lactis*, and *S. diacetylactis*. The survival rate was

Table 32
Average Biological Activities of Four Single-Strain Lactic Streptococci Stored at
—4°F. (—20°C.) and —320°F. (—196°C.)

Storage Time (Days)	Cell Viability (% Survival)		Developed Acidity (%)		Proteinase Active (%)	
	—4°F. —20°C.	—320°F. —196°C.	—4°F. —20°C.	—320°F. —196°C.	—4°F. —20°C.	—320°F. —196°C.
0	100	100	100	100	100	100
3	54	93	69	98	30	82
30	42	89	31	91	24	82
60	5	81	19	83	24	77

Source: Data averaged from those of Cowman and Speck (1965).

greater at the lower temperature, and numbers of surviving organisms decreased with storage time. It was concluded that the relative proportion of strains in mixed cultures would not remain constant under frozen storage.

Duggan *et al.* (1959) froze cultures of *L. acidophilus* at 14°F. (—10°C.), —8°F. (—22°C.), and —76°F. (—60°C.), and after 8 weeks obtained survival of 80, 82, and 88% respectively. Recent work on freezing in liquid nitrogen indicates that this lower temperature, —320°F. (—196°C.), keeps cultures viable for longer periods. Data from Cowman and Speck (1965) reproduced in Table 32 show the advantage of using liquid nitrogen.

Commercial liquid nitrogen-frozen cultures are now available for cheesemaking (Johnson *et al.* 1967). The cultures are grown in special bacteriophage inhibitory media (Hargrove *et al.* 1961), supplemented with pancreas extract to provide optimum activity (Speck *et al.* 1958), packaged in vials and frozen and stored in liquid nitrogen. The cheesemaker receives ten or more strains of cultures selected for cheesemaking characteristics. He grows the seed, mother, intermediate, and bulk starter cultures from the stock of frozen culture. This is replenished every 30 days. The cultures have retained high viability at —320°F. (—196°C.) for up to three years. Figure 67 shows the plastic vial containing the cultures and the thermal container for storage and shipment of the cultures in liquid nitrogen. This program (Johnson *et al.* 1967) appears to offer an excellent plan for the control of phage and for maintenance of peak activity in starters.

Freezing Concentrated Cultures

After growth to maximum numbers the culture may be centrifuged from the growth medium, resuspended in a protective menstruum such as milk, and placed in frozen storage. The cells should be harvested for freezing when they near the peak of their logarithmic growth phase. The bacteria are readily removed from the medium by centrifugal separation.

There has been commercial development in the production of frozen



Courtesy of V. W. Christensen, Marshall Dairy Laboratories, Inc., Madison, Wis. (1967)

Fig. 67. Liquid-nitrogen frozen culture in 1-ml. plastic vial is removed from thermal container containing liquid nitrogen

Containers and cultures are distributed to cheesemakers.

concentrated cultures. One contains the proper balance of organisms for cheese or cultured milk (Dairy Technics 1967). Its use is said to eliminate the need for mother cultures, intermediate starter, and bulk starter for buttermilk, sour cream, and even for cottage cheese made by the overnight method. However, large quantities of bulk starter are required for manufacture of cottage cheese, so the cost of using concentrated preparations in lieu of bulk starters could become prohibitive. Another concentrated culture preparation which is frozen and maintained in liquid nitrogen is ready to be added direct to the milk for bulk starter. Some 10 cultures for cheese and 12 for buttermilk and sour cream are available (Hansen 1967).

Freeze-Drying Cultures

When primary cultures are held for research and propagation they are usually freeze-dried. The range of loss in viability may vary from 10 to 90%

of the population, depending upon conditions of cultivation, pH, buffering of the media, the strain and age of the culture, and other factors. Survival rates of 60 to 70% are common with freeze-dried culture organisms when conditions are optimum.

Cultures to be freeze-dried should be grown on media most suitable for the strain used. Drying should be done when the culture has reached its peak in the logarithmic growth phase. Usually only small quantities of cultures are freeze-dried for stock and storage. Freezing should be rapid, and the drying carried to a residual moisture content of 1 to 2%.

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The Preparation for Freezing and Freezing of Eggs

HISTORICAL

The frozen egg industry began in the late 1890's when W. T. S. White and H. J. Keith independently conceived the idea of breaking the eggs from the shell and freezing the egg meats. In the early days, frozen eggs were prepared as a means of disposing of eggs that were unsuitable for sale in the shell (cracked and soiled shells). Today, the frozen egg industry utilizes the most modern equipment, practices the latest methods of plant sanitation, and breaks high-quality shell eggs. Since egg production occurs on a seasonal basis, freezing is an efficient means of conserving eggs for use throughout the year. To consistently produce top quality frozen eggs, it is essential that strict attention be paid to initial high-quality shell stock, plant sanitation, rapid rate of freezing, and holding of the frozen product at a low temperature. Freezing temperatures between -10° and -40°F . (-23° and -40°C .) are commonly practiced, with some form of rapid air circulation. The object of rapid rate of freezing is to decrease the temperature of the liquid quickly below the zone of bacterial multiplication, and thus minimize deterioration for an extended period of time.

SIZE OF THE FROZEN EGG INDUSTRY

Total liquid egg production has increased from 229 million to about 629 million pounds from 1940 to 1965 (Table 33). Liquid eggs may be used for immediate consumption, as frozen, or in the dried form. From 1900 to the outbreak of World War 2, most of the eggs broken were used in the frozen form. It is observed in Table 33 that 82.7% of the 1940 production of 229 million pounds was disposed of in the frozen form and 11.8% in the dried form. The great demands for egg products for the armed services and lend-lease during and immediately following the war years account for the large-scale production of eggs in the period from 1942 through 1950. Koudele and Heinsohn (1964) reported that about 70% of the annual liquid egg production during the war years 1942 to 1946 was dried.

From 1955 to 1965, more of the liquid egg production was used in the frozen form and for immediate consumption, and only 20 to 30% for

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